PCT/EP 00/09254
10/089028 #2

Europäisches Patentamt | European Patent Office | Office européen des brevets

PTO/SB/21

4

Bescheinigung | Certificate | Attestation

Die angehefteten Unterlagen stimmen mit der ursprünglich eingereichten Fassung der auf dem nächsten Blatt bezeichneten europäischen Patentanmeldung überein.

The attached documents are exact copies of the European patent application described on the following page, as originally filed.

Les documents fixés à cette attestation sont conformes à la version initialement déposée de la demande de brevet européen spécifiée à la page suivante.

Patentanmeldung Nr. Patent application No. Demande de brevet n°

99307444.2

PRIORITY DOCUMENT

SUBMITTED OR TRANSMITTED IN COMPLIANCE WITH RULE 17.1(a) OR (b)

Der Präsident des Europäischen Patentamts:
Im Auftrag

For the President of the European Patent Office

Le Président de l'Office européen des brevets
p.o.

I.L.C. HATTEN-HECKMAN

DEN HAAG, DEN
THE HAGUE, 21/12/00
LA HAYE, LE

Europäisches Patentamt
European Patent Office
Office européen des brevets

Blatt 2 der Bescheinigung
Sheet 2 of the certificate
Page 2 de l'attestation

Anmeldung Nr:
Application no:
Demande n°: 99307444.2

Anmeldetag
Date of filing
Date de dépôt: 21/09/99

Anmelder
Applicant(s)
Demandeur(s):
SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V.
2596 HR Den Haag
NETHERLANDS

Bezeichnung der Erfindung
Title of the invention
Titre de l'invention:
Process to remove particles from a mixture of particles and liquid

In Anspruch genommene Priorität(en) / Priority(ies) claimed / Priorité(s) revendiquée(s)

Staat: State: Pays:	Tag: Date: Date:	Aktenzeichen: File no. Numéro de dépôt:

Internationale Patentklassifikation
International Patent classification
Classification internationale des brevets:
C10J3/52

Am Anmeldetag benannte Vertragstaaten
Contracting states designated at date of filing: AT/BE/CH/CY/DE/DK/ES/FI/FR/GB/GR/IE/IT/LI/LU/MC/NL/PT/SE/TR
Etats contractants désignés lors du dépôt

Bemerkungen
Remarks
Remarques

TS 0919 EPC

Crw

PROCESS TO REMOVE PARTICLES FROM A MIXTURE OF PARTICLES AND LIQUID

The present invention is directed to a process for removing solid particles from a vessel containing a mixture of said particles and a liquid. The invention is furthermore directed to an apparatus for performing this process.

Such a process is described in EP-A-290087. In this process a mixture of water and solid slag is batchwise sluiced out of a pressurised gasification system. Liquid slag is a by-product of the gasification or partial combustion of, for example, coal. Liquid slag is drained through the outlet located at the reactor bottom and passed by gravity through a slag discharge means into a water bath or slag quenching vessel where the mixture of water and solid slag particles are formed. The batchwise sluicing of the mixture from the pressurized gasification system to a lower-pressure discharge zone is performed by means of a lockhopper. During the sluicing cycle the lockhopper is isolated from the gasification system by closing one or more valves in the connecting line between the slag quench vessel and the lockhopper.

A problem of this sluicing procedure is that, when the valve between the lockhopper and the quench vessel is closed, the slag accumulating up-stream this valve has a tendency for bridging at the narrow space just above the said valve. It has appeared very difficult to have the

slag to fall into the lockhopper after reconnecting the lockhopper to the gasification system. This problem is solved by the process disclosed in EP-A-290087, wherein a permanent nitrogen-gas bubble or nitrogen-gas cap is maintained in the lockhopper. By ensuring that the pressure of the said nitrogen gas bubble is lower than ~~the pressure in the slag quench vessel an initial~~ downwards flow of water and slag during opening of the valves between the lockhopper and the slag quench vessel is achieved.

Although the above process has proven to work satisfactory in commercial practice it still has some disadvantages. One disadvantage is the need to install additional equipment to supply nitrogen and the associated costs of the nitrogen consumption during every sluicing cycle. A further disadvantage is that together with the discharged mixture an amount of hydrogen sulfide is also discharged. Hydrogen sulfide is formed in the reactor from sulphur containing compounds which are present in the hydrocarbon feed. Part of the hydrogen sulfide will dissolve in the water present in the slag quench vessel and will thus be discharged together with the slag.

The object of the present invention is a simple process to separate solid particles from a vessel containing a mixture of said particles and a liquid.

This object is achieved by the following process. Process to remove solid particles from a vessel containing a mixture of said solid particles and a liquid, wherein a second vessel is fluidly connected to said first vessel by means of a connecting conduit,

through which conduit the solid particles are transported to the second vessel and liquid, which is poor in solids, is extracted from the second vessel and liquid which is poor in solids in supplied to the first vessel.

By performing the process according the invention it is possible to discharge solid particles from a vessel ~~containing a mixture of liquid and solid particles~~, wherein the amount of liquid being discharged is lower than in prior art processes. This is especially advantageous when used in the above described gasification process. Less hydrogen sulfide will thus be discharged together with the slag particles. The hydrogen sulfide normally discharged together with the slag will now be discharged with the synthesis product gasses. Because the synthesis gasses typically contain a certain amount of hydrogen sulfide it will be no problem to remove this additional amount of hydrogen sulfide in the existing downstream hydrogen sulfide removal sections. It has furthermore been found that the process can suitably be used in a sluicing mode of operation without particles obstructing the valves in a manner as described above. Further advantages of the present process will become apparent when reading the detailed description of this invention.

Preferably the liquid which is extracted from the second vessel is supplied to the first vessel through a second conduit. More preferably all of the liquid extracted from the second vessel is supplied to the first vessel, wherein the volume extracted from the second vessel is equal to the volume supplied to the first vessel.

Figure 1 represents an apparatus for performing the above described preferred embodiment of the process according to the present invention. The apparatus comprises a first vessel (1), a second vessel (2), preferably positioned below the first vessel, and a first (3) and second (4) conduit fluidly connecting the first and second vessel. The first conduit (3) is preferably located such that solids can move by gravity from the first vessel (1) to the second vessel (2). Second conduit (4) is provided with pumping means (5) to transport liquid from the second vessel to the first vessel. Suitable means to pump a liquid are for example a gear pump, a lobe pump, a rotary pump, a centrifugal pump or a riser. The inlet of second conduit (4) is located such that liquid which is poor in solids is pumped from the second vessel (2) into the first vessel (1). Solids entering second vessel (2) via conduit (3) will accumulate in the lower part of the second vessel resulting in that the upper part of the second vessel is poor in solids relative to the lower part. Preferably the inlet (6) of conduit (4) is therefore located in the upper part of the second vessel (2) and away from the outlet opening (8) of the first conduit (3) entering the second vessel (2). More preferably a tubular shield (7) is present around the outlet (8) of conduit (3) which directs the solids entering the second vessel (2) downwards and away from the inlet (6) of the second conduit (4). The second vessel is furthermore provided with an outlet opening (9) through which solid particles can be discharged and the first vessel is provided with an inlet opening (10) for receiving the mixture.

By extracting relatively clean liquid from the second vessel and adding relatively clean liquid to the first vessel a sort of suction is achieved which predominately pulls the solids from the first vessel to the second vessel. The second vessel must be large enough to enable the solids to settle sufficiently in order to achieve a region which is poor in solids and a lower region which is rich in solids. Solids can be continuously or discontinuously discharged from the lower part of the second vessel which part is rich in solids. The ratio of volume of liquid which is extracted from the second vessel relative to the volume of solids passing conduit (3) in the same time period is preferably between 0.7 and 1.5 and more preferably between 0.8 and 1. Most preferably the volume of liquid extracted from vessel (2) and supplied to vessel (1) is about the same as the volume of solids passing conduit (3). The liquid in conduit (3) will then be kept close to stagnant.

The present process is very advantageous to be used in a situation wherein the pressure in the first vessel is higher than the pressure of the environment into which the separated solids are discharged to. For example in a gasification process the pressure in the quench vessel being the first vessel in the process according the invention, is typically between 20 and 60 bars, while the solids are normally discharged at about ambient pressure.

Figure 2 represents an apparatus which can be used to separate particles from a vessel having a higher pressure to a lower pressure environment. The Figure illustrates an apparatus as in Figure 1 with additional valves (11, 13) present in conduits (3) and (9) in order to operate

in a sluice mode. The Figure also shows a discharge zone (12) and the lower part of a partial combustion reactor zone (14). Preferably the present process is then used in a sluice cycle mode, as for example illustrated in above cited EP-A-290087, wherein the solids are removed batchwise by alternately performing the following steps:

(a) removing solids from the first vessel (1) to the second vessel (2) as described above,

(b) closing a valve (11) present in the first conduit (3), and

(c) removing the solids from the second vessel (2) to a discharge zone (12) by opening a second valve (13) and closing said valve (13) after the solids are removed.

The process according to the invention can be used in any process to separate solid particles from a mixture. The process according the invention is preferably performed on a mixture of solids and liquid, wherein more than 80% of the solid particles in the mixture have a diameter of between $0.25\ 10^{-3}$ and $5\ 10^{-3}$ m. The type of solids and liquid is not essential except that the viscosity of the liquid under the operating conditions should be such to enable the particles to fall by gravity with an acceptable speed in such liquid. The density of the solids should be higher than the density of the liquid, preferably the particle density is more than 1.5 times the liquid density. Examples of such processes are catalyst particles present in reaction mixtures obtained in for example stirred tank reactors. The present method and apparatus provide a simple method to discharge part

or all of the catalyst from a reaction environment. The present process is especially advantageous when used to separate slag from a mixture of slag and water, which slag is formed in a process for the preparation of synthesis gas by partial combustion of finely dispersed solid carbon-containing fuel with an oxygen-containing gas. Examples of carbon-containing fuel are coal, peat, wood, coke, for example petroleum coke, soot, carbon containing waste, biomass and mixtures of these. Mixtures of the aforementioned feedstocks and metal containing waste streams can also be used as feed. It has been found that the mass flux of the slag through the first conduit (3) is preferably between 100-150 kg slag particles per square meter of the cross sectional area of the conduit or valve, whatever the smallest, per second ($kg/m^2/s$).

The invention shall be illustrated by the following non-limiting Examples.

Example 1

In an experimental set-up as illustrated in Figure 2 vessel 1 was loaded with a mixture of water and 172 kg slag obtained from a coal gasification process having a density of 2335 kg/m^3. Most slag particles were present at the bottom of vessel 1 near valve 11. Vessel 2 was loaded with clean water. After opening of valve 11, having a diameter of 10 cm, and after start-up of pump 5 a stable sluicing flow through the valve was observed. The pump flow rate was 15.5 litre/minute and the 172 kg of slag was sluiced out in 3.65 minutes. The ratio of volume of liquid which is transported from the second vessel to the first vessel via conduit (4) relative to

the volume of solids passing conduit (3) in the same time period is in this example thus 0.75.

Example 2

Example 1 was repeated except that the pump flow was 36.3 litre/minute. The same 172 kg of slag was sluiced out in 2.44 minutes. The ratio of volume of liquid which is transported from the second vessel to the first vessel via conduit (4) relative to the volume of solids passing conduit (3) in the same time period is in this example 1.18.

Comparative experiment A

Example 1 was repeated except that pump 5 was not used. The flow through valve 11 was very unstable and the experiment was repeated 15 times in order to obtain a reliable test result. On average it took the 172 kg of slag 5.6 minutes to pass valve 11.

The above experimental results show that by using the process according to the invention a high flow of slag can be transported from an upper vessel to a lower vessel while minimizing the amount of water being transported from said upper vessel to said lower vessel.

C L A I M S

1. Process to remove solid particles from a vessel containing a mixture of said solid particles and a liquid, wherein a second vessel is fluidly connected to said first vessel by means of a connecting conduit, through which conduit the solid particles are transported to the second vessel; liquid, which is poor in solids, is extracted from the second vessel; and liquid, which is poor in solids in supplied to the first vessel.

2. Process according to claim 1, wherein the liquid which is extracted from the second vessel is supplied to the first vessel through a second conduit.

3. Process according to any one of claims 1-2, wherein the second vessel is located below the first vessel.

4. Process according to any one of claims 1-3, wherein the liquid, which is poor in solids, is discharged from the upper part of the second vessel at a position away from the inlet opening of the first conduit entering the second vessel.

5. Process according to any one of claims 1-4, wherein the ratio of volume of liquid which is extracted from the second vessel relative to the volume of solids particles being transported to the second vessel in the same time period is between 0.7 and 1.5.

6. Process according to claim 5, wherein the ratio is between 0.8 and 1.

7. Process to remove solid particles from a first vessel to a second vessel, wherein the solids are removed

batchwise by alternately performing the following steps:
(a) removing solids from the first vessel to the second vessel in the manner as claimed in any one of claims 1-6,
(b) closing a valve present in the first conduit, and
(c) removing the solids from the second vessel to a discharge zone by opening a second valve and closing said valve after the solids are removed.

8. Process according to claim 7, wherein the pressure in the first vessel is higher than the pressure of the discharge zone.

9. Process according to any one of claims 1-8, wherein the liquid/solids mixture is obtained by contacting liquid slag and water, which liquid slag is formed in a process for the preparation of synthesis gas by partial combustion of finely dispersed solid carbon-containing fuel with an oxygen-containing gas.

10. Apparatus to remove solid particles from a vessel containing a mixture of said solid particles and a liquid comprising said vessel, hereinafter referred to as the first vessel, a second vessel positioned below the first vessel, a first and second conduit fluidly connecting the first and second vessel, wherein the first conduit is located such that solids can move by gravity from the first vessel to the second vessel and wherein the second conduit is provided with means to pump liquid from the second vessel to the first vessel, wherein the inlet opening of the second conduit is located such that liquid which is poor in solids is pumped from the second vessel into the first vessel.

C45/TS0919PD

TS 0919 EPC

A B S T R A C T

PROCESS TO REMOVE PARTICLES FROM A MIXTURE OF PARTICLES AND LIQUID

Process to remove solid particles from a vessel containing a mixture of said solid particles and a liquid, wherein a second vessel is fluidly connected to said first vessel by means of a connecting conduit, through which conduit the solid particles are transported to the second vessel and liquid, which is poor in solids, is transported through a second conduit from the second vessel to the first vessel.

C45/TS0919PD

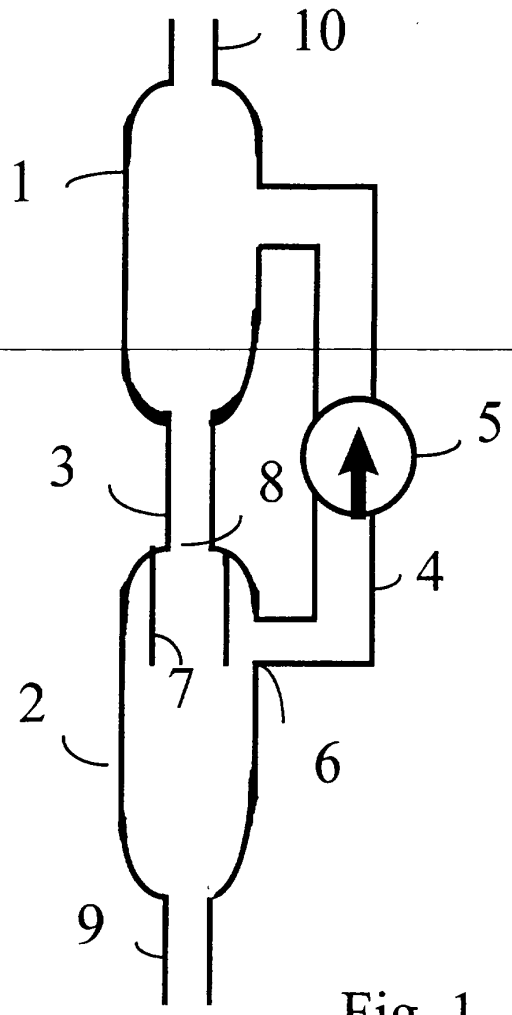

Fig. 1

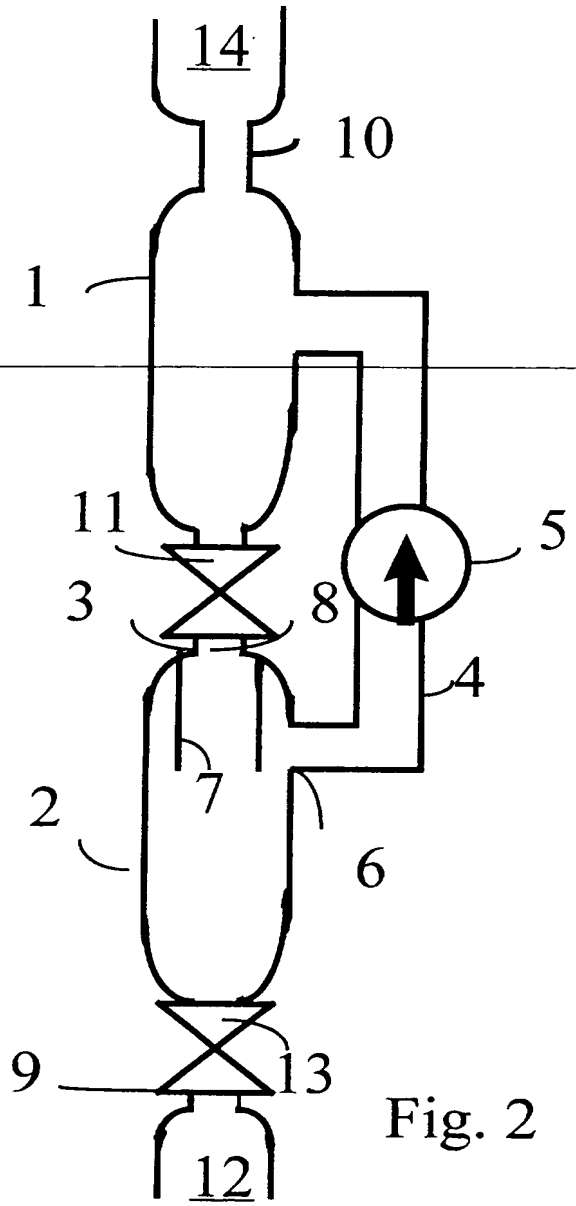

Fig. 2